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REVIEW ARTICLE

PHYSICO-CHEMICAL CHARACTERISATIONS OF ALUM SLUDGES FROM THE DRINKING WATER PRODUCTION PLANT OF SINÉMATIALI, NORTHEN CÔTE D'IVOIRE

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ABSTRACT

In the northern region of Côte d'Ivoire, alum sludges from the Sinématiali drinking water production plantare directlyrejected into the natural environment without any specific treatment.This constitutes a source of pollution and potential health hazards. The aim of this study is to evaluate the potentialvalorisation of these alum sludges in construction materials by carryingout physico-chemical characterisation tests. Chemical, mineralogical, spectral, microscopic and wettability studies were performed on these alum sludges. The results of the chemical composition through inductively coupled plasma optical emission spectrometry (ICP-OES) analysis shows that the most abundant oxides in alum sludges are silica (SiO_2), alumina (Al_2O_3) and iron oxide (Fe_2O_3) with respective proportions of 50.80%, 36.57% and 10.32%. Potassium oxide (K_2O), sodiumoxide (Na_2O) and titanium oxide (TiO_2) are onlypresent in small quantities. Mineralogical analysis by X-ray diffraction (XRD) reveals that the alum sludges are mainly composed of poorly crystallised kaolinite (78%). Minorphases such as hematite (9.3%), illite (7.2%) and quartz (4.5%) were also detected. Infrared spectroscopy reveals the presence of absorption bands at 688.778 and 743 cm^{-1}, 1007, 1013 and 1077 cm^{-1} characteristics of clays in kaolinite form. Microscopic observation shows a microporous and amorphous structure of these clayey sludges. The high value of water demand (1133 µL/g) favors a good reactivity of these sludges in aqueous solution. These characterisations indicate that alum sludges of Sinématiali are highly suitable for incorporation with Portland cement in the production of refractory construction bricks, as well as mortars and reinforced concretes.

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INTRODUCTION

Water is a source of life (Hoedeman *et al*., 2010). Indeed, all human activity involves water consumption, whether domestic, industrial, medicinal, artisanal or agricultural level. In north of Côte d'Ivoire, only 20% of the population have access to drinking water (Kassi *et al.*, 2020). This reveals a major problem. To address this issue, the Ivorian Government launched a project aimed at improving the supply of drinking water in urban areas. The objective of this project is not only to enhance the quality of drinking water but also to increase its production accessibility in the northern towns such as Korhogo and Ferkessédougou. The town of Sinématiali has also benefited of this project through the establishment of a drinking water production facility. In general, the production of drinking water generates enormous quantities of wastes known as alum sludges (NSandji *et al.*, 2023). In most African countries and particularly in Côte d'Ivoire, nearly 100% of the sludges produced are directly discharged into the natural environment without any specific treatment. This practice is a significant source of pollution and potential health risks (NSandji *et al*, 2023). The Sinématiali drinking water production plant also faces this challenge related to the management and disposal of alum sludges. Recycling or valorizing these sludges represents a potential solution to conserve natural resources and reduce environmental pressure.-Therefore, in order to address the issue of the alum sludges produced by the drinking water treatment station in the town of Sinématiali, located in the north of Côte d'Ivoire, study was carried out to evaluate their potential valorisation in construction materials through physico-chemical characterisation texts.

MATERIALS AND METHODS

Presentation of the site: The sub-prefecture of Sinématiali (Figure 1) is located in the northern part of Côte d'Ivoire in the Poro region. It lies approximately 592 km from Abidjan and about 35 km from the town of Korhogo, covering an area of 680 km². Its geographical coordinates are: 09°27'37.1"' N and 005°38'83.2"' W, with an average altitude of 342 m. This town is served by the Tine and Tielogo rivers, as well as the White Bandama river. The Sinématiali drinking water production station supplies potable water to the towns of Sinématiali, Korhogo and Ferkessédougou. The raw water is drawn from the White Bandama river and ransported to the treatment facility, where it undergoes several purification processes to make it suitable for human consumption

Sampling: The sample of red-brown alum sludge was collected using a plastic jar in a liquid form after purging the station's decanter. The sample was transported to the laboratory, where it was oven-dried at

110°C for 24 hours to remove moisture. At the end of this process, a dry red-brown powder was obtained for subsequent analyses (Figure 2).

Methods of analysis: The chemical analysis of the alum sludge powder obtained was performed using inductively coupled plasma optical emission spectroscopy (ICP-OES). A 40 mg portion of the dried powder, previously ground to a particle size of 100 µm using an agate mortar, was weighted and placed in a teflon digestion vessel. Subsequently, 4 ml of hydrofluoric acid (HF, 28% volumes) and 2 ml of nitric acid (HNO_3, 68%volumes) were added. The mixture was subjected to microwave-assisted digestion for 50 minutes using an ANTON Paar Pro Multiwave system. The resulting solution was analyzed with an Optima 8300 DV ICP-OES spectrometer - Perkin Elmer. The mineralogical analysis of alum sludges was conducted using a D8 diffractometer (Brucker) equipped with a Bragg-Brentano montage. A portion of the finely ground sludge powder was introduced into the diffractometer and irradiated with X-ray generated by the diffractometer. Crystal phase identification was carried out with EVA (Brucker) software using reference data from the Powder Diffraction File (PDF) database of the International Centre for Diffraction Data (ICDD).

The alum sludge powder was mixed with 10 g of finely ground potassium bromide (KBr) and pressed into pellets before being placed on the diamond crystal of the instrument for spectral acquisitions. Spectral acquisitions were performed in the range of 500-4000 cm^{-1}, with 64 scans and a resolution of 4 cm^{-1}.The data were then analysed using OMNIC software (Nicolet instrument). Microstructural characterization of the powder was conducted using Scanning Electron Microscopy (SEM). This technique allows the observation of the surface topography of the powder by scanning its surface with an electron beam and collecting the resulting image. For sample preparation, a small quantity of alum sludge powder was suspended in distilled water. After ultrasonic agitation for 5 minutes, a drop of each suspension was deposited onto a sample holder and allowed to dry. The sample holder was then placed in the SEM chamber for observation. The apparatus used wasa field emission scanning electron microscope FEI Quanta FEG 450. The water demand of alum powder, expressed as wettability (µL/g), corresponds to the volume of water adsorbed by one gram of powder until saturation. The procedure involves weighing 1.00 g of alum sludge powder into a glass cup, then gradually adding distilled water in 10 µL increments using a micropipette, until visual saturation of the granular mixture was achieved.

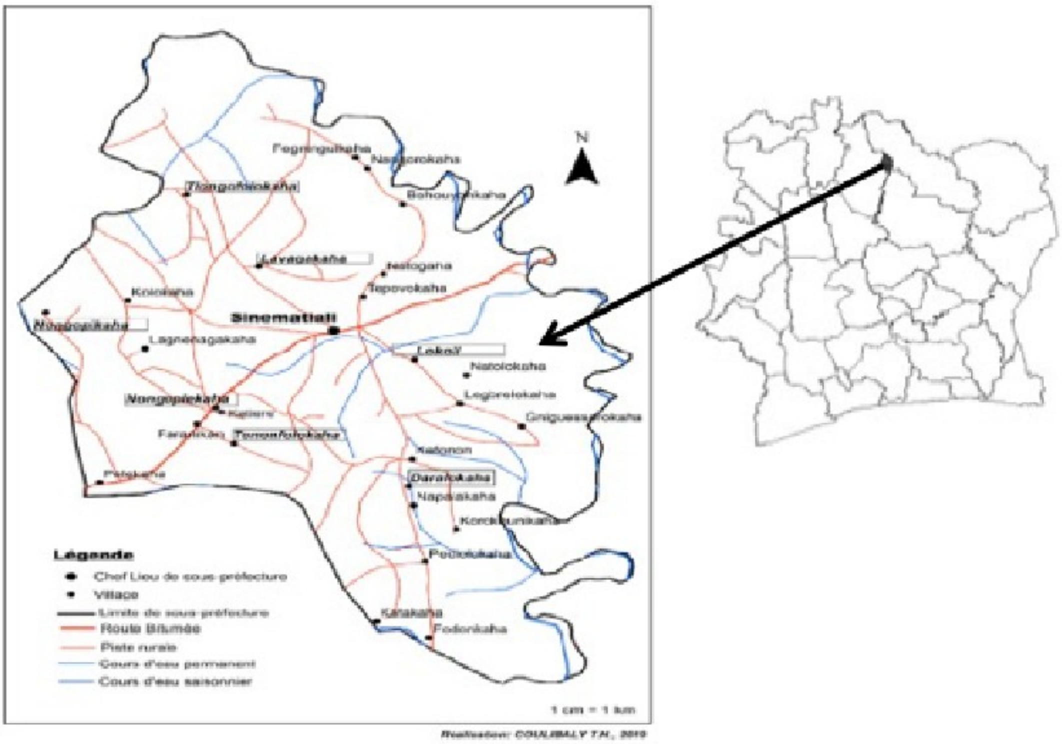


Figure 1 .Sub-prefecture of Sinématiali

The functional groups present in the alum sludges were determined by Fourier transform infrared spectroscopy (FT-IR) using the Thermoficher Scientific Nicolet 380 spectrometer.

Figure 2. Physical aspect of Sinématiali alum sludge dried at 110°C

RESULTS AND DISCUSSION

Results

Chemical analysis: Table 1 presents the results of the chemical composition of the alum sludges collected from Sinématiali, as determined by ICP-OES. The measured abundances are expressed as oxide percentages, relative to the mass of the sample dried at 110 °C. The results indicate that silica (SiO_2) and alumina (Al_2O_3) are the major oxides present, with respective contents of 50.80% and 36.57%. This means that these sludges are aluminosilicate materials with a SiO_2/Al_2O_3 mass ratio of 1.4 (with a corresponding molar ratio of 1.2). These findings are consistent with those reported by other researchers (Dahhou *et al.*, 2012 ; Emerouwa *et al.*, 2008). A relatively high content of iron oxide (Fe_2O_3, 10.32 %) is also observed, indicating that these sludges are iron-rich aluminosilicates. The elevated iron content is likely responsible for the red-brown coloration of the samples. Titanium oxide (TiO_2) and potassium oxide (K_2O) are present in minor quantities, while sodium oxide (Na_2O) is detected in even smaller amounts. Due to their low alkali flux (K_2O and Na_2O) content, these alum sludges exhibit favorable properties for use as raw materials in refractory product manufacturing (Ledoussa, 1985). The sum of the content of the three main oxides, namely SiO_2 + Al_2O_3 + Fe_2O_3, is 97.7%.

Mineralogical analysis

X-rays diffraction (XRD): The mineralogical composition of the alum sludge powder was determined from the X-ray diffraction. The corresponding X-ray diffractogram is shown in Figure 3. The diffraction pattern reveals that kaolinite ($KAl_2(Si_3Al)O_{10}(OH)_2$) is the dominant mineral phase, as indicated by its characteristic diffraction peaks. In addition, the presence of hematite (Fe_2O_3) and illite (K_2O, $2H_2O$, $2Al_2(Si_3Al)O_{10}OH$) is also observed. These phases are associated with minor amounts of quartz (SiO_2) and Rutile (TiO_2).

Fourier transform infrared spectral analysis: The result of the infrared (IR) spectral analysis of the dried sludge sample is shown in Figure 4. The spectrum reveals the presence of several characteristic functional groups. The absorption bands observed at 3612 cm^{-1} and 3691.24 cm^{-1} correspond to the stretching vibrations of structural –OH groups, while the bands at 1605 cm^{-1} is attributed to the bending vibration of molecular water (H_2O).

The bands located at 1007 cm^{-1}; 1013.45 cm^{-1}; 1013 cm^{-1} and 1077 cm^{-1} are assigned to Si-O and Si-O-Si stretching vibrations, typical of silicate frameworks. Furthermore, the absorption bands around 780 cm^{-1} and 540 cm^{-1} are characteristics of aluminosilicate minerals, such as kaolinite or illite. Overall, the IR spectrum confirms the information provided by X-ray diffraction.

Microscopic observations by SEM: Figure 5 shows scanning electron microscope (SEM) images of the dried alum sludge sample observed at magnifications of 2 µm and 5 µm. The microstructure reveals platelet-shaped particles with irregular contours, typical of kaolinite-type morphologies. These platelets are stacked and agglomerated, forming clusters that indicate a partially disordered structure. This morphology is consistent with that commonly observed in poorly crystallised kaolinite and illite minerals of (Goure-Doubi, 2013), which is characteristic of the presence of an amorphous phase. These observations are in agreement with previously observed results. The presence of this amorphous phase confers pozzolanic reactivity to the alum sludge, suggesting its potential use as a supplementary cementitious material or as a precursor in geopolymer formulations (Mounjouohou *et al.*, 2019).

Wettability: The average water demand of the dried alum sludge sample was determined to be 1133 µL/g. Such a high wettability value (≥ 760 µL/g), suggests that the alum sludge exhibits strong reactivity in aqueous media. Furthermore, the presence of an amorphous phase, as evidenced by SEM and XRD analyses, would increase the pozzolanic activity of these kaolinite-based alum sludges (Gharzouni *et al.*, 2017).

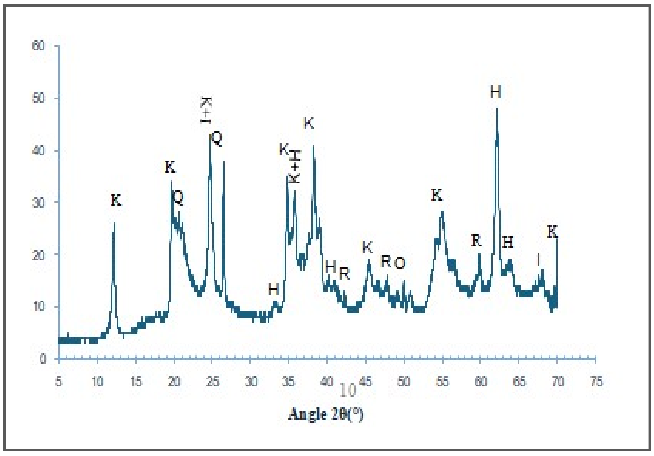


Figure 3. XRD pattern of the studied alum sludge

Table 1. Chemical composition of the studied alum sludge

Name of oxides	Chemical elements in oxides	Mass percentage (%)	
silica	SiO_2	50,80	SiO_2/Al_2O_3 =1,4
Aluminate	Al_2O_3	36,57	
Iron oxide	Fe_2O_3	10,32	
Potassium oxide	K_2O	1,04	$SiO_2+Al_2O_3+Fe_2O_3$=97,7%
Sodium oxide	Na_2O	0,13	
Titanium dioxide	TiO_2	1,14	

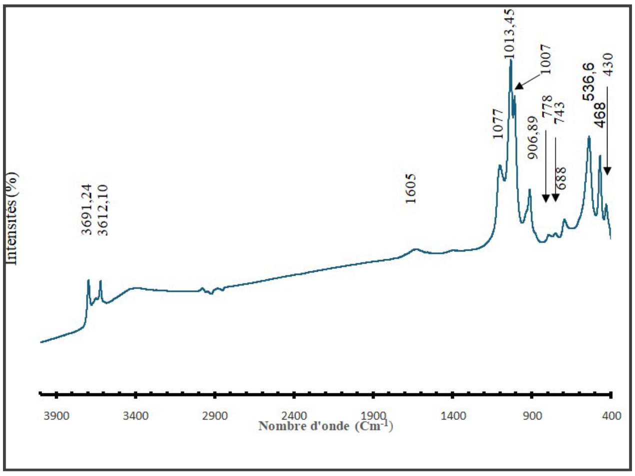
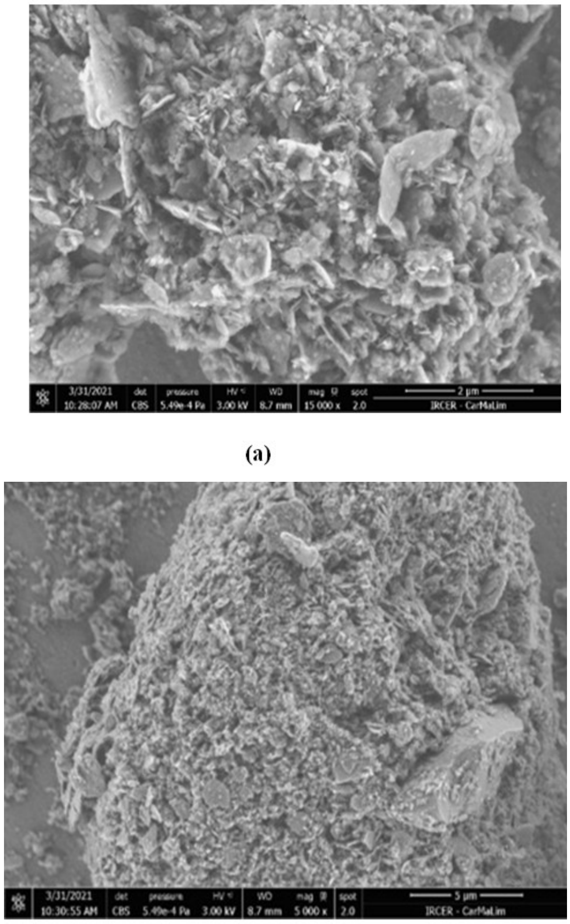


Figure 4. Infrared spectrum of the studied alum sludges



(a)

(b)

Figure 5. SEM micrographs showing the microstructure of the dried alum sludge sample at magnifications of 2 µm (a) and 5 µm (b)

DISCUSSION

The ICP-OES analysis conducted on the alum sludges collected from the drinking water treatment plant in Sinématiali reveals that the main constituents are silica (SiO_2, 50.8%), alumina (Al_2O_3, 36.57%) and iron oxide (Fe_2O_3, 10.32%). The high alumina content is likely related to the use of aluminate-based coagulant or flocculants (alkaline or halide forms) in the drinking water treatment process. The silica-to-alumina ratio, estimated at 1.4 (Table 1), serves as a useful indicator of the relative abundance of clay minerals. This value suggests that the studied sludges are rich in clay minerals (Bodian *et al.*, 2021). Furthermore, the combined content of the three main oxides, namely $SiO_2+Al_2O_3+Fe_2O_3$, is 97.7%, suggesting a possible pozzolanic activity of the dried sludges in accordance with the American standard (ASTM C618-12a, 2012). These findings are consistent with the XDR results, which revealed the predominant presence of kaolinite and illite, associated with smaller amounts of hematite and quartz. These kaolinitic clays probably originate from sedimentary deposit present at the wastewater discharge site (Kaga *et al*., 2024). Due to their high kaolinite content, these sludges could be valorised in cementitious materials and/or geopolymers (Mounjouohou *et al.*, 2019). The relatively low quartz content (approximately 5%) may further support their use as a natural colouring agent in the manufacture of earthenware and porcelain (Emerouwa *et al.*, 2008).

Moreover, the low alkali content of this type of material suggests its potential suitability as a raw material for refractory product manufacturing (Ledoussa, 1985). Indeed, kaolinite, as a clay mineral, is widely used and valued not only in the production of common ceramics but also in technical and high-performance ceramics (Emerouwa *et al.*, 2008). The incorporation of materials with pozzolanic properties such as metakaolin obtained from the calcination of kaolin, can be envisaged as a partial replacement (5-15%) of Portland cement in mortars and concretes. These pozzolanic materials react with portlandite ($Ca(OH)_2$) to form hydrated compounds similar in composition and structure to those produced by cement hydration (Zamble *et al*., 2025 ; Tognonvi *et al*., 2022). The infrared spectral analysis mainly reveals the absorption bands characteristics of silica and alumina, which together form the structural framework of clay mineral in kaolinite form. These results are in good agreement with the findings of the chemical and mineralogical analyses, confirming the dominant presence of aluminosilicate phases in the studied alum sludge. Microscopic observations of the sludge sample reveal that it consists of poorly crystallised kaolinite clays, justifying the presence of an amorphous phase (Goure Doubi, 2013). This characteristic supports the classification of the studied sludge as a pozzolanic material, suitable for valorisation in cementitious and geopolymer formulation (Mounjouohou *et al.*, 2019). In fact, a pozzolan is defined as a siliceous or silico-aluminous material that possesses little or no intrinsic binding capacity, but which, when finely ground and in the presence of water, can chemically react with calcium hydroxide ($Ca(OH)_2$) under ambient temperature and pressure to form a binder. The porous and amorphous structure of such materials enhance both their water absorption capacity, and their pozzolanic reactivity (Gharzouni *et al.*, 2017). The wettability of the studied sludge sample was measured at 1133 µL/g. This high-water demand (≥ 760 µL/g) indicates strong reactivity in aqueous solution, confirming its pozzolanic potential.

CONCLUSION

The objective of this study was to characterise the alum sludges discharged from the Sinématiali drinking water treatment plant in order to explore their potential for valorization. Chemical characterization revealed that Sinématiali alum sludges are mainly composed of three major oxides: silica (SiO_2), alumina (Al_2O_3) and iron oxide (Fe_2O_3) with respective proportions of 50.80%, 36.57% and 10.32%. The predominance of these three oxides indicates that the material consists of iron-rich aluminosilicates with low alkali flux content (K_2O and Na_2O), confirming its clayey nature. FTIR analysis confirmed that the alum sludges are primarily composed of kaolinite-type clays. SEM observations revealed a microporous and amorphous structures, characteristics of poorly crystallized clay materials. Such morphology supports their classification as as pozzolanic materials. The wettability test showed a high-water demand of 1133 µL/g, exceeding the threshold value (≥ 760 µL/g), which reflects strong reactivity in aqueous media. Overall, the results demonstrate that the Sinématiali alum sludges possess significant pozzolanic potential and can be partially combined with Portland cement or geopolymers. They show promise for use in the manufacture of refractory bricks, as well as in the production of mortars and reinforced concretes, thereby contributing to sustainable waste valorization and resource-efficient construction materials. Further studies should focus on the pozzolanic characterization of the Sinématiali alum sludges through tests such as the Frattini and Chapelle methods, as well as compressive strength measurements on cement–sludge blends at various curing ages. Complementary analyses using XRD, FTIR, and TGA/DTA would also help confirm the formation of hydrated pozzolanic phases and validate the potential of these sludges as sustainable supplementary cementitious materials.

Key points

- Alum sludge is an aluminosilicate rich in iron.
- Material possessing pozzolanic properties.
- Material that can be valorized in cementitious or geopolymer materials
- Alum sludge has good reactivity in aqueous solution.

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